

ELECTRON MICROSCOPY OF NATURAL AND EPITAXIAL DIAMOND

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Semiconducting diamond films have the potential for use as a material in which to build active electronic devices capable of operating at high temperatures or in high radiation environments. Ultimately, it is preferable to use low-defect-density single crystal diamond for device fabrication. We have previously investigated polycrystalline diamond films with transmission electron microscopy (TEM) and scanning electron microscopy (SEM)^{e.g.1}, and homoepitaxial films with SEM-based techniques^{e.g.2}. This contribution describes some of our most recent observations of the microstructure of natural diamond single crystals and homoepitaxial diamond thin films using TEM.

We are developing techniques to thin diamonds to electron transparency in the region of interest; particularly as diamond is difficult to mechanically thin. One simple (but time consuming) approach for the fabrication of plan-view diamond TEM samples is to thin only by ion milling. Figs. 1 and 2 show TEM micrographs taken from a (100) natural type IIb (semiconducting) diamond crystal and a (100) natural type Ia diamond that have been ion milled to electron transparency. The mottled background contrast observed in both images is believed to be an artifact due to ion milling. An average dislocation density of $\sim 5 \times 10^7 \text{ cm}^{-2}$ was observed in the type IIb material, and the dislocation distribution was found to be nonuniform. Dislocations were not observed via TEM in the type Ia diamond (dislocation density $< 10^5 \text{ cm}^{-2}$), but nitrogen-containing platelets that lie on {100} planes, characteristic of type Ia diamond, were observed. A technique for more rapid thinning for plan-view TEM sample preparation was achieved by laser ablation (Fig. 3), which is described in more detail elsewhere³. Although it is recognized that defect densities will vary from stone to stone for natural diamond, type Ia has consistently been found to show lower dislocation densities.

Homoepitaxial diamond films were grown on (100) type Ia diamond substrates using water/methanol mixtures in an rf-driven plasma-enhanced chemical vapor deposition (PECVD) reactor. The growth conditions were comparable to that described elsewhere⁴. They were then ion milled from the back-side for plan-view TEM examination. Figs. 4 and 5 respectively show the featureless topography recorded by a field emission gun SEM and dislocations observed by TEM in a film grown for 50 minutes. The average dislocation density was found to be $\sim 5 \times 10^6 \text{ cm}^{-2}$, and no other types of defects were observed. This dislocation density would be considered high by single crystal Si standards, but it does represent the current benchmark for diamond epitaxy. We are certain we are observing the epilayer because of the absence of nitrogen-containing platelets. Note also that the micro-Raman spectrum of the diamond epitaxial layer showed the characteristic LO phonon peak with a FWHM of 2.4 cm^{-1} , comparable to that of the substrate. These results show that growth of homoepitaxial diamond can be achieved on natural type Ia diamond to a reasonable standard. In other words, the presence of the nitrogen-containing platelets in the substrate does not result in catastrophic dislocation densities in the diamond epilayer.⁵

References

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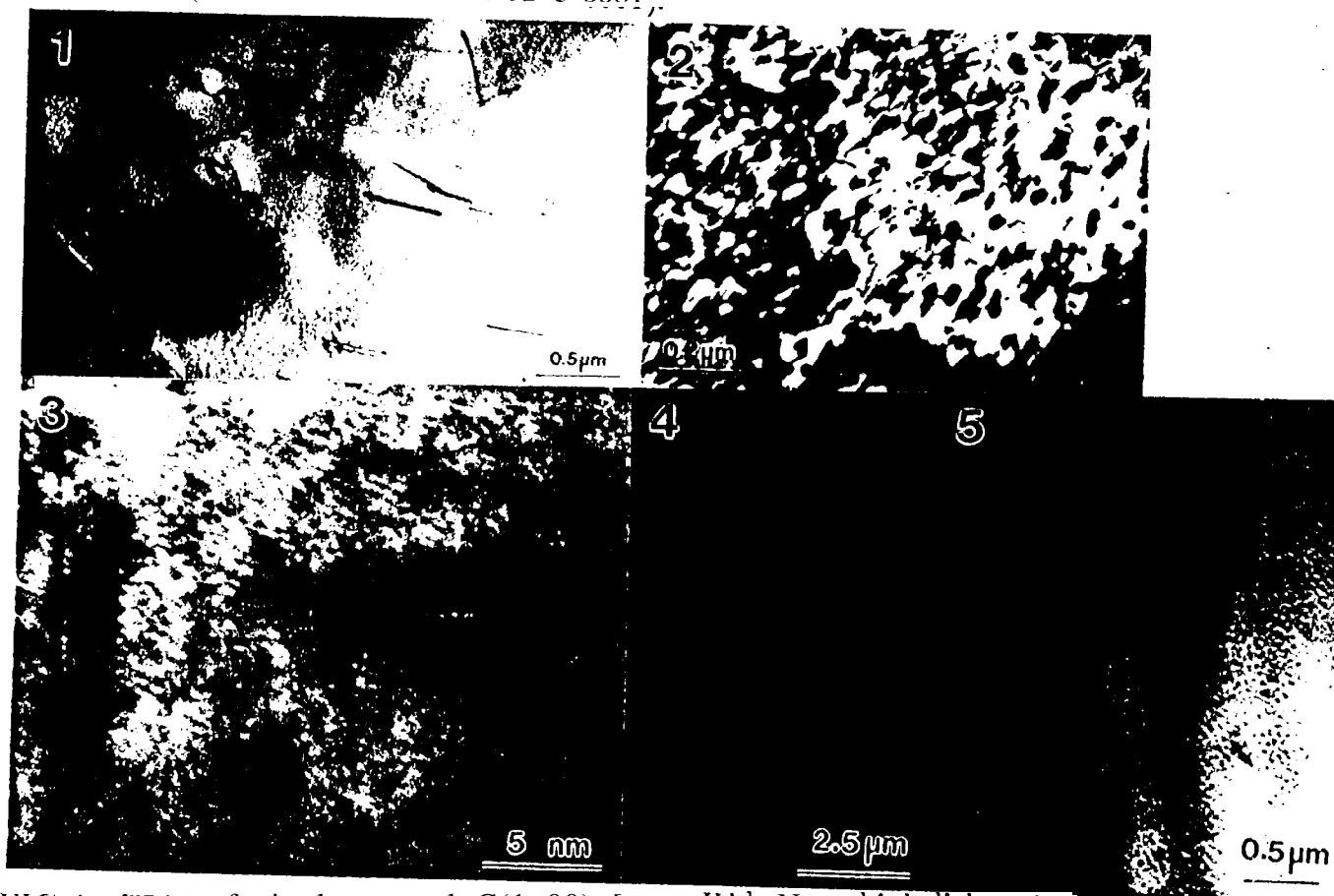


FIG. 1.--TEM of single crystal C(1 00) [type IIb]. Note high dislocation density. Sample prepared by ion milling.

FIG. 2.--TEM of single crystal C(1 00) [type Ia]. Note the presence of {100} nitrogen platelets (shown inclined in this [1 10] orientation) characteristic of natural type Ia diamond. Sample prepared by ion milling.

FIG. 3.--Higher magnification TEM of single crystal C(100) [type Ia] in [1 00] orientation. Sample prepared by laser ablation.

FIG. 4.--SEM topographical image of diamond homoepitaxial film grown using water/methanol mixture. The surface is featureless.

FIG. 5.--TEM plan-view of diamond homoepitaxial film grown using water/methanol mixture. Sample prepared by ion milling.